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(54) Title: NONAQUEOUS DRAG REDUCING SUSPENSIONS

(57) Abstract

This invention relates to improving the flow of flowing streams of hydrocarbons, such as petroleum or finished products, in a conduit such as a pipeline. The present invention also relates to a stable, nonagglomerating nonaqueous suspension suitable for improving flow of hydrocarbons in conduits, particularly finished product hydrocarbons in conduits.

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NONAOUEOUS DRAG REDUCING SUSPENSIONS Field of the Invention

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This invention relates to improvement of flow of hydrocarbons, whether crude oil or refined products, in constricted conduits such as pipelines. The invention finds application both in science and industry, such as the oil industry.

It is well known in the art that certain polymers which are oil soluble may be polymerized in the presence of produce high molecular catalysts to weight, crystalline, hydrocarbon soluble polymers by various means. These polymers when dissolved in a hydrocarbon fluid flowing through a conduit greatly reduce turbulent flow and decrease "drag", thus reducing the amount of horsepower needed to move a given volume of hydrocarbon, or conversely enable greater volumes of fluid to be moved with a given In short, these polymers are drag amount of power. reducers or flow improvers for hydrocarbon fluids.

It is of particular interest to note that the high molecular weight polymers known as drag reducers are used in very dilute solutions ranging up to about 100 parts per million in the hydrocarbon flowing through the conduit. These materials display useful flow characteristics not present in the commonly known crystalline, largely nonhydrocarbon soluble artifact-forming polymers such polypropylene. polyethylene and In contrast, the hydrocarbon soluble materials of the present invention are noted for their effectiveness as anti-misting agents and drag reducing agents.

As is known in the art, these polymers are susceptible to shear degradation when dissolved in the hydrocarbons in which drag is being reduced. Thus passage

through a pump, severe constrictions in a conduit or the like which produce excessive turbulent flow leads to degradation of the polymer, which thereafter has reduced effectiveness. Consequently, it is important that these materials be placed into the flowing hydrocarbon stream in a form which achieves certain desirable features.

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First, the polymer should be placed in a form adequate for easy transportation and handling without exotic or unusual equipment, since injection points for the polymer into the flowing hydrocarbon stream can often be at remote and inaccessible locations and require the movement of material to such locations. Secondly, the polymer must be in a form which dissolves rapidly in the hydrocarbon being transported, since the polymers have little drag reducing effect until solubilized into the hydrocarbon stream. Third, the hydrocarbon should also be innocuous to the ultimate purpose of the hydrocarbon fluid. example, in the case of crude oil flowing through a pipeline, larger amounts of water and contaminants can be tolerated than in finished pipeline products such as diesel fuel, gasoline, and jet fuel which are ultimately destined to be burned in internal combustion engines and turbine engines.

Currently, a number of different commercial approaches are being taken to the problem of preparing, dissolving, transporting and using such drag reducing polymers. In use, the polymers form extremely dilute solutions ranging from about 1 to up to about 100 parts per million polymer and hydrocarbon, yet remain effective in order to receive drag reduction or anti-misting. A common commercial method is to prepare the polymer in dilute solutions in an inert solvent such as kerosene or other solvating material as set forth in Mack, U.S. Patent 4,433,123. In this reference, Mack utilizes a solution of

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high molecular weight polymer suitable for use as a drag reducing agent when produced by polymerization of alpha olefins in a hydrocarbon solvent. The entire mixture, containing polyolefin, solvent, and catalyst particles is used without separation to form dilute solutions of the polymer in crude oil or finished hydrocarbons. one disadvantage of such approach is the use of a solvent, which poses a shipping and handling difficulty and may constitute a hazard. In addition, the product itself forms a gel-like substance which is difficult to introduce into flowing hydrocarbon streams and which becomes extremely viscous and difficult to handle under cold weather temperature conditions, such as when injection conduits at remote locations is required.

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A second, more recent approach is to prepare the polymers as a solid material as described in patents issued to Fenton, U.S. Patent No. 4,584,244, and to O'Mara, U.S. Patent Nos. 4,720,397; 4,826,728; and 4,837,249; all of which deal with finely grinding drag reducing polymers to a very small particle size in an inert atmosphere below the glass transition point of the polymers and in the presence of a partitioning agent to form a multi-layered coating which holds the inert atmosphere adjacent to the particle polymers after grinding. The O'Mara patents teach it is mandatory that the reduction to fine particle size be carried out in inert atmosphere utilizing a refrigerant such as liquid nitrogen, and a coating agent while grinding the polymer to an appropriate size, where the coating agent prevents contact of oxygen with the freshly ground polymer particles.

Another recent method is set forth in U.S. Patents 5,449,732 and 5,504,132 which deal with preparing a hydrocarbon-soluble drag reducing aqueous suspension or water alcohol suspension. The patents also teach that bulk

polymerization of the poly alphaolefins produces a material which is much higher in molecular weight and which provides reduction drag as compared to polymerization, where molecular weight declines as polymer That invention solved the problem of content increases. such high molecular producing weight alkenes commercially satisfactory manner for use in drag reducing materials and further, provided methods for suspending such materials in an aqueous or alcohol/aqueous system.

However, these prior art processes have distinct problems, including clumping, and conversion to a solid in the aqueous suspension in extremely warm temperature conditions. In addition, it is necessary to protect the suspension against freezing in extremely cold temperature conditions.

Description of the Prior Art

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U.S. Patent No. 5,244,937 deals with stable suspensions nonagglomerating agueous of oil polymeric friction reducers by suspending such materials in water, together with a water-soluble thickening agent in a mixture of water or water and alcohol. U.S. Patents 5,449,732; 5,504,131; and 5,504,132 all deal with a method of preparing ultrahigh molecular weight poly alphaolefins using a bulk polymerization system, cryogrinding in the presence of a partitioning agent, and suspending the resulting mixture in a water or water-alcohol mixture. U.S. Patent No. 5,539,044 deals with the aqueous suspension of drag reducing polymers together with a surfactant having a hydrophilic/lipophilic balance (HLB) in a particular range to enhance suspension and durability of suspension.

All of the prior art processes have certain drawbacks and unwanted features, including agglomeration at high temperatures, freezing at cold temperatures, and the

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necessity of adding an aqueous-based material into a hydrocarbon flowing stream. Such addition is not particularly troublesome in crude oil pipelines in view of the small amount injected, which generally becomes lost in the overall pipeline water content, but such materials can be objectionable in finished product pipelines even at low concentrations, as the presence of water may force the product outside specified limits.

Consequently, it would be a great advantage to provide a nonaqueous suspension of highly effective drag reducing materials for use in hydrocarbons flowing through conduits.

Summary of the Invention

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This invention relates to improving the flow of flowing streams of hydrocarbons, such as petroleum or finished products, in a conduit, such as a pipeline. The present invention also relates to a stable, nonagglomerating nonaqueous suspension suitable for improving flow of hydrocarbons in conduits, particularly finished product hydrocarbons in conduits.

The present invention comprises a method forming a heat stable, nonagueous suspension of a solid hydrocarbon soluble polyolefin friction reducing agent formed from olefins containing from 2 to 30 carbon atoms and capable of reducing drag in hydrocarbons flowing through conduits comprising (a) finely dividing the polyolefin friction reducing agent in the presence of a partitioning to agent provide free flowing polyalphaolefin material coated with said partitioning agent, and (b) dispersing the coated polyalphaolefin particles in a substantially nonaqueous suspending medium selected from the group consisting of alcohols containing 14 or less carbon atoms and glycols containing 14 or less dipropylene glycol methyl carbon atoms and

tripropylene glycol methyl ether, tetrapropylene glycol methyl ether, or ethyl ethers of similar nature, wherein the partitioning agent is a fatty acid wax. Mixtures of glycol ethers can be used as suspending agents.

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finely divided non-crystalline ultrahigh molecular weight hydrocarbon soluble polyalphaolefin can be prepared by polymerizing an olefin and reducing the polyolefin thus formed to a finely divided state at temperatures below the glass temperature of the solid drag reducing polyalphaolefin. Various methods of preparing such materials are described in Mack, U.S. Patent No. 4,433,123, where the material is formed in an inert solvent, subsequently extracted from the solvent and then cryoground. Another method of preparing an even higher molecular weight material is found in U.S. Patent No. 5,504,132 which shows the production under bulk polymerization conditions of such materials in order to achieve high molecular weights and then grinding the resulting material.

The suspension can be made in alcohols containing 14 or less carbon atoms and glycols containing 14 or less carbon atoms, or mixtures of these. Suspensions can also be made with dipropylene glycol methyl ether, tripropylene glycol methyl ether, tetrapropylene glycol methyl ether, mixtures ethers, and of these. "substantially free of water" is used to indicate that incidental amounts of water may be present, but do not rise to the level of providing suspension capabilities to the drag reducing suspension. Thus, the amounts of the water present are generally below 1 to 2 percent. The present invention provides a drag-reducing suspension by dispersing finely ground polymer particles throughout the non-aqueous suspending medium. Suspensions differ from the solutions Mack as described in 4,433,123 in that discrete

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particles are present. Thus, even if the suspending medium is a partial solvent, the present invention retains discrete particles in suspension. In some embodiments, the very fine particles originally cryoground may swell or clump to a greater or lesser extent, but the suspending medium is selected or formulated to maintain discrete particles of various size and provides for such particles to be solvent-swollen where appropriate. In general, the suspending medium is a non-solvent with respect to the polymer, or has a solubility so low with respect to the polymer particles that discrete particles are maintained. Generally, the shorter the suspending medium backbone chain, the less the solubility.

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During cryogrinding of the polymer, the presence of a fatty acid wax is necessary in order to make a drag reducing suspension, with saturated fatty acid waxes preferred for the most stable drag reducing suspensions. Use of other materials as partitioning agents such as carbon blacks, or talc for example, appear to cause separation and instability of the system over long periods of time. In addition, the type of partitioning agent used will directly effect the choice of the suspending agent used. For example, the alcohol chain length, regardless of whether the alcohol is branched or linear can become longer with certain partitioning agents. Still other partitioning agents prove to be incapable of forming a suspension with any nonaqueous material tested, and consequently are unsuitable for use in the present invention.

The present invention provides significant advantages over previous water-based suspensions in that the system has freeze protection, permits a higher weight percent of active polymer in the slurry, eliminates agglomeration and heat instability problems, eliminates concerns about adding water to product pipelines, and is a

simple process to carry out, not requiring surfactants and other complicated stability systems.

Detailed Description of the Invention

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The suspending medium of the present invention can be an alcohol, whether linear or branched, either alone or in combination with a polyethylene glycol containing up to 14 carbon atoms. While not critical to this invention, it is generally preferred that the majority of the alcohol suspending agent be a branched alcohol, as to provide alcohols seem some slight advantage maintaining suspension stability. In general, alcohols up to 14 carbon atoms and polyethylene glycols of up to 14 carbon atoms can be used. However, a more preferred are alcohols and polyethylene combination containing up to 10 carbon atoms. A most preferred suspending agent contains alcohols and polyethylene glycols containing up to 8 carbon atoms. Suspensions can also be made with dipropylene glycol methyl ether, tripropylene glycol methyl ether, tetrapropylene glycol methyl ether, ethyl glycol ethers and mixtures of these. Of course, it will be realized by those skilled in the art that various mixtures of these various carbon atom length alcohols and polyethylene glycols and glycol ethers can be used to provide a "tailored" suspending medium for the particular polyolefin loading and service conditions. In particular, it should be noted that a particular mixture can vary depending upon basic concepts such as price, convenience or availability, as well as technical guestions of stability, solubility, long-term storage, and compatibility with the flowing hydrocarbon.

Partitioning agents used when finely dividing the polyalphaolefin drag reducer are fatty acid waxes, and preferably are saturated fatty acid waxes for maximum stability. Use of other partitioning agents such as

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caltium stearate, and other metal stearates can be tolerated in a minor amount, but it is necessary that the weight of non-fatty acid wax partitioning agents be in a distinct minority (less than one fourth the total volume of partitioning agents) since they tend to form agglomeration products at high temperatures in nonaqueous systems. The most preferred partitioning materials (partitioning agents) are bis stearamides, primary stearamides, secondary stearamides, and mixtures of these.

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Representative examples of effective materials are primary stearamides, generally a C_{16} - C_{20} saturated acid reacted with NH₃; secondary stearamides, which chemically are C_{16} - C_{20} saturated acids reacted with NH₃ to form amines, then further reacting C_{16} - C_{20} saturated acids with the amine to form stearamides; and bis-stearamides, which are C_{16} - C_{20} saturated acids reacted with diamine to form stearamides.

Partitioning agents which are most effective in nonaqueous systems are those having a saturated acid portion. Other materials which are incompletely reacted and contain unsaturated portions of the acid molecule are generally less favorable since they tend to form solid materials upon extreme heating or higher temperatures for extended times when in the presence of the non-aqueous suspending medium. The most preferred partitioning agents for long-term stability are stearamide and bis-stearamide.

In addition, depending upon the combination of alcohols used, it may be desirable to utilize a thickening agent such as hydroxy propyl cellulose to stabilize the suspension. Such thickening agents will generally comprise 1.5 percent by weight or less of the suspension.

The present invention permits solids loading in nonaqueous suspension of up to 80 percent by weight, producing paste-like consistencies using the most preferred

partitioning agents, while the drag-reducing polymer will still immediately dissolve and reduce drag in hydrocarbons effectively.

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Examples of alcohols useful in the practice of the present invention include linear and branched alcohols containing up to 14 carbon atoms. In a preferred mode, the alcohols will contain up to 10 carbon atoms and will in addition be combined with a glycol to prepare a suspending medium of the proper density. Generally, a ratio of alcohol to glycol will range from about 100 percent to 0 percent to about 50 percent to 50 percent respectively, although such ratios are not critical. In alcohols having densities which are near the density of the coated, finely ground polyalphaolefin are preferred. Other examples of effective suspending mediums are dipropylene glycol methyl ether, tripropylene glycol methyl ether, tetrapropylene glycol methyl ether, or ethyl ethers of similar nature. Mixtures of these suspending mediums can be used.

The concentration of solids in the suspending medium can be up to about 80 percent by weight. For freer flowing materials, the concentration of solids will generally be somewhat lower, about 30 to 60 percent by weight. Lower concentrations can be used, but are not as economically effective for transporting suspensions over long distances to injection points in hydrocarbon pipeline transportation systems.

The present invention is more concretely described with reference to the examples below, in which all parts and percentages are by weight, unless otherwise specified. The examples are provided only to illustrate the present invention and not to limit it.

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EXAMPLE 1

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Α non-crystalline, high molecular weight hydrocarbon-soluble polymer is cryoground to a finely divided state in the presence of 30 percent by weight of ethylene bis-stearamide partitioning agent to produce a non-agglomerating, free-flowing powder. The powder is added to a non-aqueous suspending medium comprised of isooctyl alcohol (80 percent by weight) and propylene glycol (20 percent by weight) to form a suspension containing 45 percent solids by weight (polymer and coating agent) to form latex-like suspension having free-flowing characteristics. Long-term separation stability enhanced by adding in addition 0.15 percent by weight, on total suspension weight, of hydroxypropyl cellulose under agitation. The resulting suspension is capable of impacting drag reduction to hydrocarbon flowing The suspension remains stable at 105°F through conduits. for a period of five weeks.

EXAMPLE 2

non-crystalline, Α high molecular hydrocarbon-soluble polymer is cryoground to a divided state in the presence of 30 percent by weight of ethylene bis-stearamide partitioning agent to produce a non-agglomerating, free-flowing powder. The powder is added to a non-aqueous suspending medium comprised of dipropylene glycol methyl ether to form a suspension containing 50 percent solids by weight (polymer and coating agent) to form a latex-like suspension having free-flowing characteristics. Long-term separation stability enhanced by adding in addition 0.15 percent by weight, on total suspension weight, of hydroxypropyl based cellulos under agitation. The resulting suspension is capable of impacting drag reduction to hydrocarbon flowing through conduits. The suspension remains stable at 105°F for a period of five weeks.

EXAMPLE 3

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A non-crystalline, high molecular weight hydrocarbon-soluble drag reducing polydecene was cryoground to a finely divided state, in a cryogenic mill, in the presence of 30 percent by weight of each of the following coating agents in the total powder created, respectively:

Stearyl stearamide (secondary amide - alkyl and amide both saturated)

Oleyl palmitamide (secondary amide - alkyl is unsaturated; amide is saturated)

Ethylene bis-oleamide (bis-amide - amides are unsaturated)

Erucamide (primary amide - amide is unsaturated)

Stearamide (primary amide - amide is saturated)

Ethylene bis-stearamide (bis-amide - amides are saturated)

The powders created from these processes were then allowed to warm back to room temperature and evaluated for their handling and/or free-flowing nature after one day period.

All the powders could be handled and maintained in a free-flowing state in order to further process by adding to suspending agents to create slurries. There were differences, however, in the "stickiness" of the powders, and their tendency, under pressure, to re-agglomerate. The best two powders from that standpoint (low stickiness, no reagglomeration) were those created with the stearamide, and with the bis-stearamide. The least attractive were those created with the bis-oleamide, oleyl palmitamide, and

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stearyl erucamide as they showed some tendency to reagglomerate if squeezed by hand.

EXAMPLE 4

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The powders from Example 3 were further processed into non-aqueous suspensions by adding these powders to a suspending medium consisting of 80% iso-octyl alcohol/20% propylene glycol by weight. The powders were added at a level to create suspensions consisting of 45% by weight These suspensions were created by simply dumping the powders into the suspending medium and stirring for several minutes. The resulting suspensions were observed in the lab over a one-month period for their stability and The stearamide coated powder, and bisflowability. stearamide coated suspensions remained fluid and stable, with no appreciable change, over the one-month period (and Samples subjected to warmer temperature (approximately 100°F) also remained fluid and stable. stearyl stearamide coated suspension remained fluid at ambient lab conditions, but a sample subjected to warmer temperature did change to a non-pourable state, over The bis-oleamide and erucamide coated several days. suspensions became very viscous over the first 24 hours and changed to a non-pouring state within a few days. oleyl palmitamide coated suspension changed to a nonpouring state within the first 24 hours. All appeared to be viable and effective drag reducing suspensions, while in a pourable state.

EXAMPLE 5

Two thousand gallons of non-aqueous drag reducing suspension was created by cryogenically grinding a non-crystalline, high molecular weight polydecene to a finely divided state in the presence of 30% by weight of ethylene

bis-stearamide in the total powder created to produce a non-agglomerating, free-flowing powder. This powder was then mixed into a suspending fluid medium consisting of iso-octyl alcohol, propylene glycol, and hydroxypropyl cellulose to create a final suspension consisting of:

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45.0% drag reducing powder

44.0% iso-octyl alcohol

11.0% propylene glycol

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0.05% hydroxypropyl cellulose

This suspension was shipped to an arctic climate (in a non-heated, non-agitated container) and injected into a 185 mile-long segment of a 47-inch I.D. pipeline carrying crude oil to effect drag reduction. At a 25 ppm, by volume, injection rate of the suspension, over 50% average drag reduction was achieved over the entire 185-mile distance of test pipeline. The remaining suspension was kept outdoors in the arctic climate for over 2 months, after which further injection tests were conducted. With ambient temperatures below -20°F, the suspension remained fluid and pumpable, and provided drag reduction in crude oil.

While certain embodiments and details have been shown for the purpose of illustrating this invention, it will be apparent to those skilled in this art that various changes and modifications may be made herein without departing from the spirit or the scope of the invention.

We claim:

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Claim 1. A method of forming a heat stable, nonaqueous suspension of a solid hydrocarbon soluble polyolefin friction reducing agent formed from olefins containing from 2 to 30 carbon atoms and capable of reducing drag in hydrocarbon flowing through conduits, comprising:

- (a) finely dividing said solid polyolefin friction reducing agent in the presence of a partitioning agent to provide a free flowing, polyalphaolefin material coated with said partitioning agent,
- (b) dispersing the coated polyalphaolefin particles in a substantially nonaqueous suspending medium selected from the group consisting of alcohols containing 14 or less carbon atoms, glycols and glycol-ethers,

wherein the partitioning agent is a fatty acid wax.

- Claim 2. A method as described in Claim 1 wherein the fatty acid wax is a saturated fatty acid wax.
- Claim 3. A method as described in Claim 2 wherein the fatty acid wax is selected from the group consisting of bis stearamides, primary stearamides, secondary stearamides, and mixtures of these.
- Claim 4. A method as described in Claim 3 wherein the saturated fatty acid wax is stearamide or bis-stearamide or mixtures of these.
- Claim 5. A method as described in Claim 4 wherein in addition a thickening agent is added.
 - Claim 6. A method as described in Claim 4 wherein the

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majority of the alcohol suspending agent is a branched alcohol.

Claim 7. A method as described in Claim 4 wherein the solids loading of the suspension is up to 80% by weight.

Claim 8. A method as described in Claim 1 wherein the glycol-ether suspending agent is at least one glycol ether selected from the group consisting of dipropylene glycol methyl ether, tripropylene glycol methyl ether, tetrapropylene glycol methyl ether, or mixtures of these.

Claim 9. A nonaqueous, heat stable, non-agglomerating suspension capable of reducing drag in hydrocarbons flowing through conduits, comprising:

- (a) a finely divided solid polyolefin friction reducing agent formed from olefins containing from 2 to 30 carbon atoms,
- (b) a fatty acid wax partitioning agent,
- (c) a suspending agent selected from the group consisting of alcohols containing 14 or fewer carbon atoms and glycols containing fewer than 14 carbon atoms, and glycol-ethers.
- 25 Claim 10. A composition as described in Claim 9 wherein the fatty acid waxes are saturated fatty acid waxes.
- Claim 11. A composition as described in Claim 10
 wherein the saturated fatty acid wax is stearamide, bisstearamide or mixtures of these.
 - Claim 12. A composition as described in Claim 11 wherein in addition a thickening agent is added.

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Claim 13. A composition as described in Claim 12 wherein the thickening agent is hydroxypropylcellulose.

Claim 14. A composition as described in Claim 11 wherein the suspending material comprises primarily branched alcohols.

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- Claim 15. A composition as described in Claim 11 wherein the suspending material comprises dipropylene glycol methyl ether, tripropylene glycol methyl ether, tetrapropylene glycol methyl ether, or mixtures of these.
- Claim 16. A composition as described in Claim 11 wherein the solids loading of the suspension is up to 80 percent of total suspension weight.

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A. CLASSIFICATION OF SUBJECT MATTER IPC(6) :CO8L 91/06; C08J 5/20								
US CL :524/275, 312 According to International Patent Classification (IPC) or to both national classification and IPC								
B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols)								
U.S.: 524/275, 312								
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched								
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)								
C. DOCUMENTS CONSIDERED TO BE RELEVANT								
Category*	Citation of document, with indication, where a	ppropriate, of the relevant passages	Relevant to claim N .					
Y	US 5,504,132 A (SMITH et al.) 02 A	1-16						
A	US 5,045,588 A (ARRANAGA) 03 S	1-16						
A	US 4,771,800 A (POMEROY) 20 Sep	1-16						
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Further documents are listed in the continuation of Box C. See patent family annex.								
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